STRUCTURE OF DOMAINS IN THIN FILM SUPERCONDUCTORS

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Submitted in partial fulfillment of
the requirements for the degree of

MASTER OF SCIENCE
IN
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ABSTRACT

Attempts were made to study intermediate state domain structure in thin indium films by application of fine diamagnetic and ferromagnetic powders. Approximate film dimensions were width 500 microns and thickness 1000 angstroms. Photographs of domain structure were sought.

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1. Introduction.

Shortly after he had succeeded in liquefying helium and while investigating the temperature dependence of electrical resistance in the newly available temperature range below 4.2°K, H. Kamerlingh-Onnes in 1911 discovered that certain metals appear to lose instantaneously all electrical resistance when cooled below some critical temperature. This instantaneous and complete loss of resistance was not anticipated by any theories then in vogue. And, although most metallic properties have since been qualitatively explained by wave-mechanical arguments, theoretical physicists are yet unable to satisfactorily account for many aspects of this phenomenon of superconductivity.

Superconductivity is shown by some 23 elements, 73 compounds, and 45 alloys. The transition or critical temperature, characteristic for each element, has been found to vary from the low of 0.35°K for hafnium to about 8°K for niobium. Some alloys and compounds have higher transition temperatures. Alloys and compounds display certain irregularities in properties; e. g., they exhibit erratic magnetic behavior and a one to two degree temperature interval over which transition occurs compared to a transition interval of one one-hundredth of a degree or less for elements. Superconducting elements lie generally towards the middle of the periodic table, there being none with either one or six valence electrons. All isotopes of these elements are them-

selves superconductors with each isotope having its own transition temperature.

Though not fully understood, many characteristics of the superconducting state and the transitions between the resistive and superconducting states have been experimentally observed. For example, one fundamental property of a superconductor is the absence of magnetic flux within the medium in its superconducting state regardless of conditions existing prior to attainment of this state. There is actually slight field penetration at the surface of the superconductor, but this field is thought to decay exponentially according to the relation $H = H_0 e^{-\frac{x}{\lambda}}$ where λ is the penetration depth and x is the distance into the specimen. (For temperature dependence curves and sample values of the penetration depth, see D. Shoenberg, Superconductivity, Sec. 5.2; Cambridge University Press, 1952). Further, as superconductivity is destroyed by an externally imposed magnetic field or passage of current through the specimen, the material transforms from its superconducting state through an intermediate state to its fully resistive state. In the intermediate state resistive and superconductive domains separated by sharply defined phase boundaries coexist. It is known that the critical or transition temperature is greatly influenced by the presence and orientation of an external magnetic field as well as the passage of current through the specimen - the greater being the magnetic field or current, the lower being

the transition temperature.[1]

The intermediate state, its causes and properties, has been and continues to be the subject of intensive research inasmuch as an appreciation of the nature of domain structure and interphase boundary energy considerations is important to the understanding of superconductivity itself. Domain structure has been displayed by application of magnetic powders to the surface of superconductors of divers configurations other than thin films and the results reported by various workers.[2-6]

The intent of this project was to study intermediate state domain structure by application of fine niobium or nickel powder to the surface of thin superconducting indium films, and observe any patterns resulting from powder particle displacement as partial transition is induced by a dc current passed through the specimen. Niobium powder, having

[1] Graphical indication of transition temperature dependence on current and magnetic field may be found in A. I. Shal'nikov and L. A. Feigin, Soviet Physics, "Doklady," 3-1, 377 (1956), and D. Shoenberg, Superconductivity, pp. 224-225; Cambridge University Press, 1952, respectively.

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[3] A. G. Meshkovsky and A. I. Shal'nikov, J. Phys. (U.S.S.R.) 11, 1, (1947).

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[6] B. M. Balashova and I. V. Sharvin, Soviet Physics, "JEPT" 4, 54 (1957)

a transition temperature of about 8^{o}K even under the influence of a few hundred gauss magnetic field, is itself a superconductor at liquid helium temperatures and is therefore perfectly diamagnetic at those temperatures. Nickel, a non-superconductor, on the other hand is a ferromagnetic material. One, then, would expect niobium particles to desert resistive domains for superconducting regions and vice versa for nickel, thereby providing the domain patterns sought. It was anticipated that a low strength magnetic field would enhance the particle displacement.

Results of experimentation with thin indium films by Doctors E. C. Crittenden, Jr., and J. N. Cooper of this laboratory indicate that there are two temperature intervals in which the intermediate state can be readily induced. Figure 1, a representation of the type of data obtained by Doctors Crittenden and Cooper, shows one such interval immediately below the transition temperature and the other immediately below the lambda point for liquid helium (2.186^{o}K). At temperatures without these two intervals (and above about 1.5^{o}K) it appears that the current necessary to initiate transition is also sufficient to complete transition.

It is considered possible that in thin rectangular films the intermediate state owes its existence to localized switching induced by supercritical current densities being developed in the vicinity of some film imperfection. A nick or other imperfection in the film might have the effect of reducing the cross sectional area of the film at a particular point,

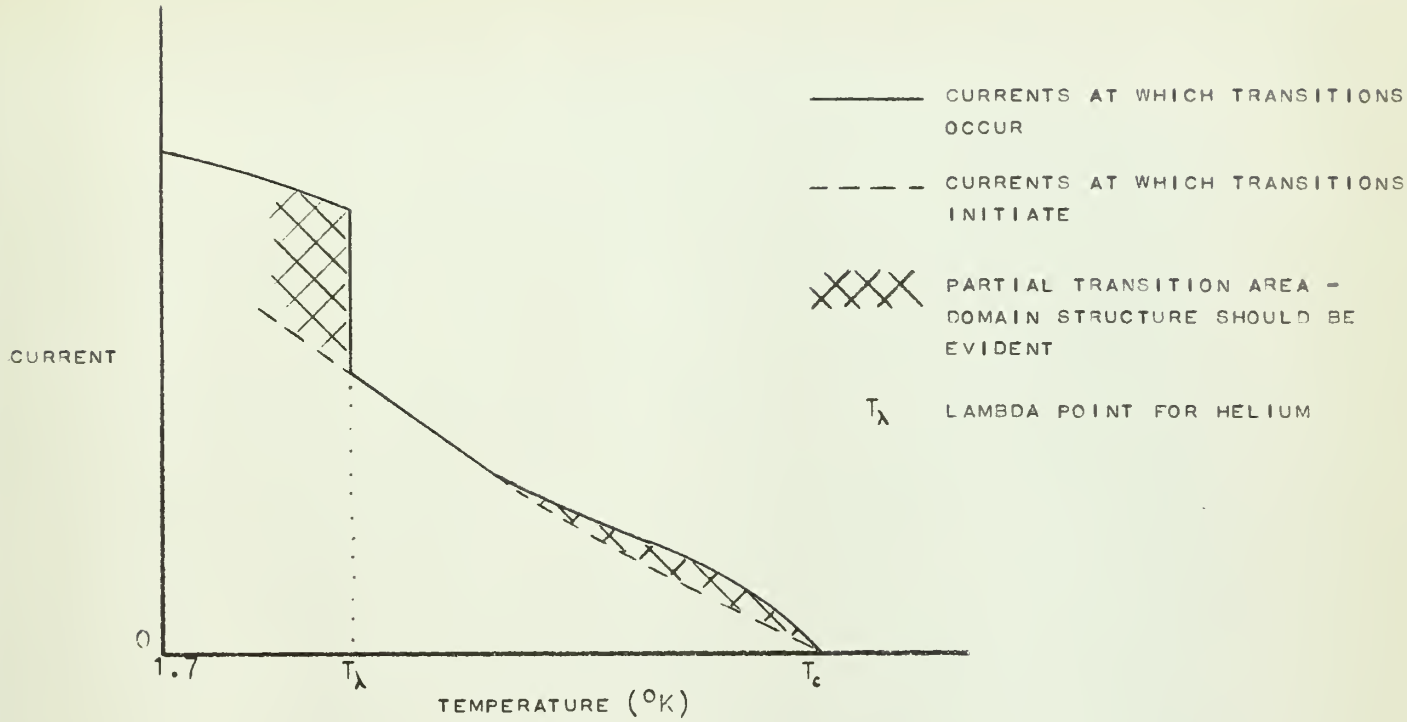


FIGURE 1 CRITICAL CURRENT DEPENDENCE ON TEMPERATURE

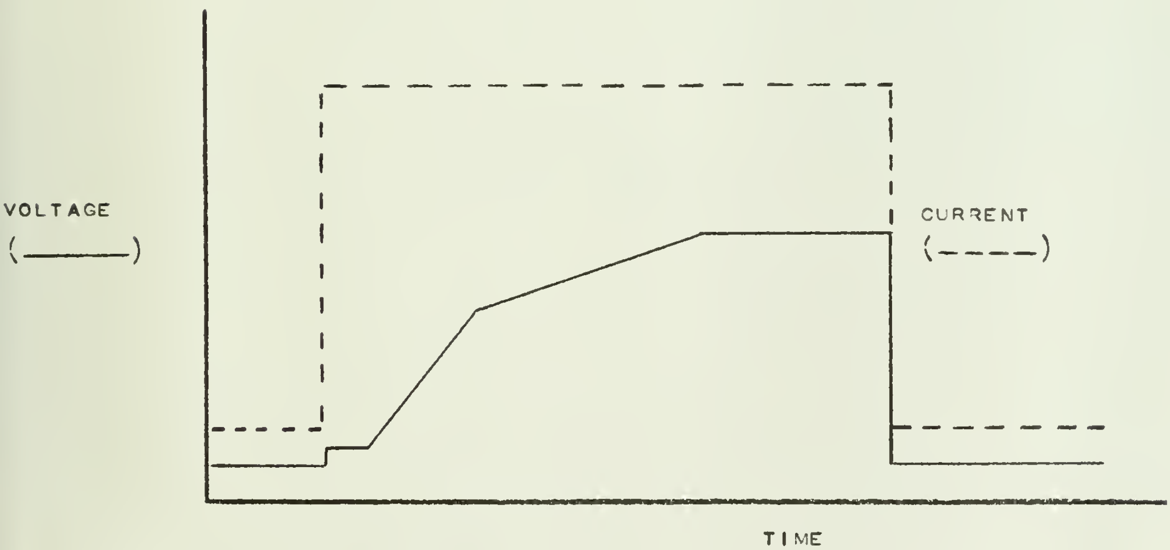


FIGURE 2 VOLTAGE RESPONSE TO CURRENT PULSE

thereby increasing the current density from a generally subcritical to a locally supercritical value.

Figure 2 is a representation of further data obtained from thin film resistance measurements by Doctors Crittenden and Cooper. The upper (dashed) trace represents a supercritical current pulse through an initially superconducting specimen. The lower trace indicates the increase of voltage along the specimen with time. Such a voltage-time relationship with two distinct slopes lends support to the supposition that, due perhaps to some imperfection, a small portion of the film first transforms and the boundaries of this resistive domain advance at a uniform rate along the film. The abrupt change in slope of the voltage-time trace would indicate that one boundary reached the end of the film before the other. Voltage-time traces similar to Figure 2 but having more than two distinct slopes might indicate that the film switched in more than one spot initially.

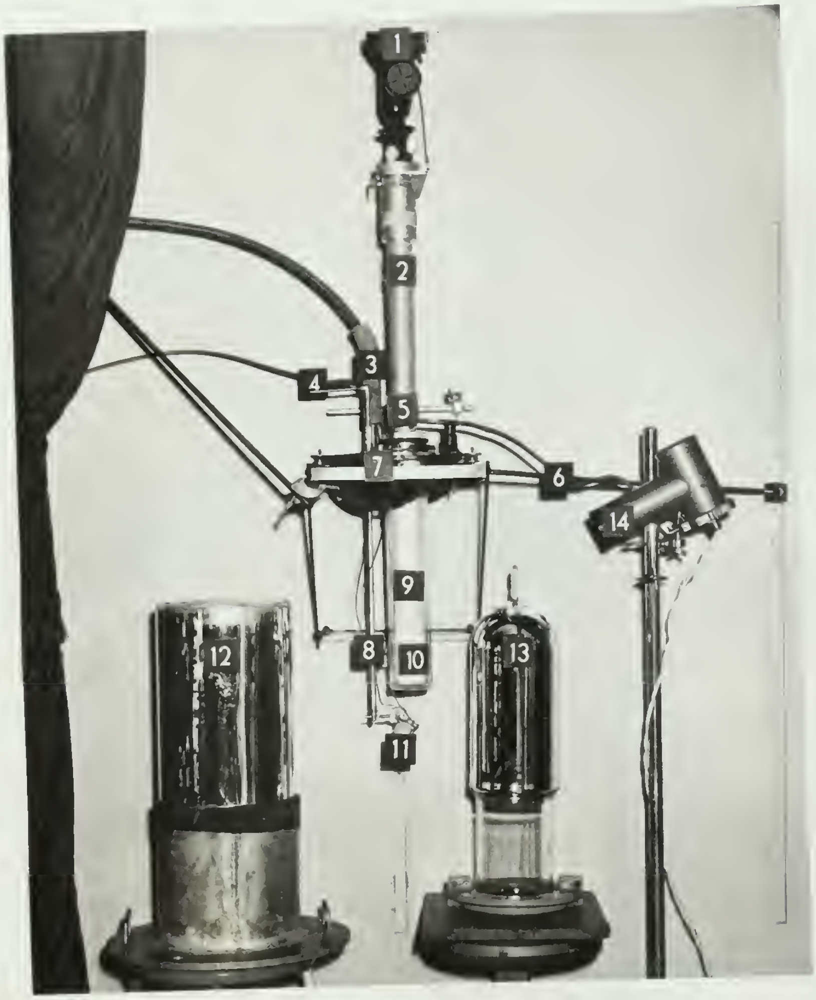
Photographs were to be made of films in the intermediate state in the expectation of observing powder patterns indicative of domain structure.

2. Experimental Procedure.

The equipment used in the project was designed to provide a means of maintaining a thin film superconductor in its intermediate state and photograph its domain structure. The experimental apparatus, partially disassembled, is pictured in Figure 3. The assembled cryostat (optical system removed) is illustrated by Figure 4. The specimen to be investigated and photographed is placed in the holder and the light path aligned as illustrated in Figure 5. The six inch dewar is then placed in position and liquid air transferred into the dewar for precooling. While the six inch dewar is precooling, the outer ten inch dewar is positioned and filled with liquid air. Upon completion of precooling, the inner dewar of the cryostat is emptied by pressurized, cooled helium gas.

Immediately following the above operation liquid helium is transferred, again by cooled helium gas. Following liquid helium transfer the metallic powder being used is introduced into the inner dewar flask. The vacuum line is then opened and temperature is lowered to the operating range. The quantities of liquid air and liquid helium required for an operating time of about 90 minutes are shown in Table I.

FIGURE 3



COMPONENTS

1 FILM PLATE ADAPTER
2 OPTICAL COLUMN
3 VACUUM TUBE
4 MANOMETER TUBE
5 POWDER INDUCTION
6 HELIUM GAS LINE
7 CRYOSTAT BASE PLATE
8 ELECTRICAL LEADS
9 3 IN. OPTICAL DEWAR
10 LENS
11 SPECIMEN HOLDER
12 10 IN. OUTER DEWAR
13 6 IN. INNER DEWAR
14 PROJECTION LAMP

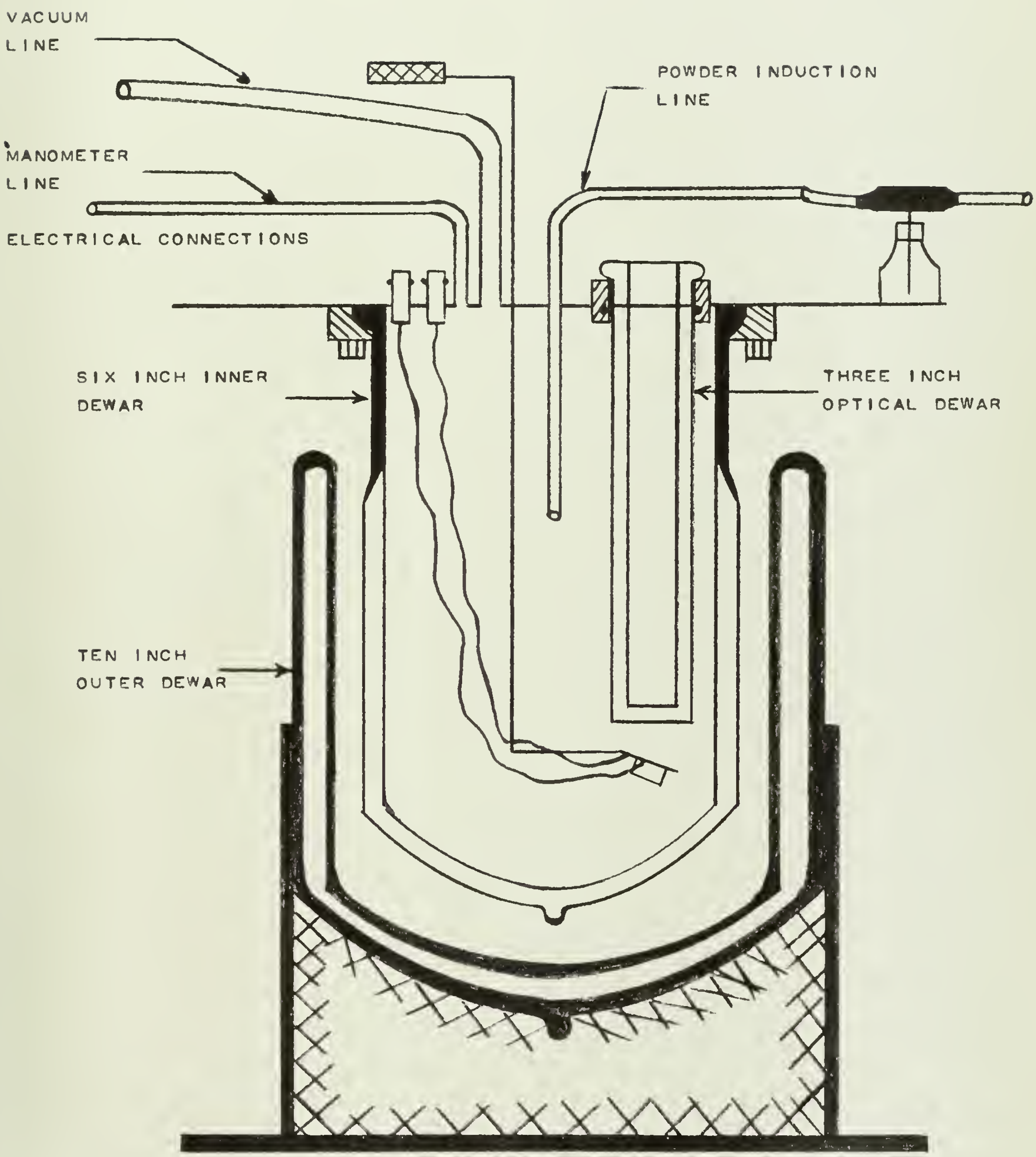


FIGURE 4 ASSEMBLED CRYOSTAT (OPTICAL SYSTEM REMOVED)

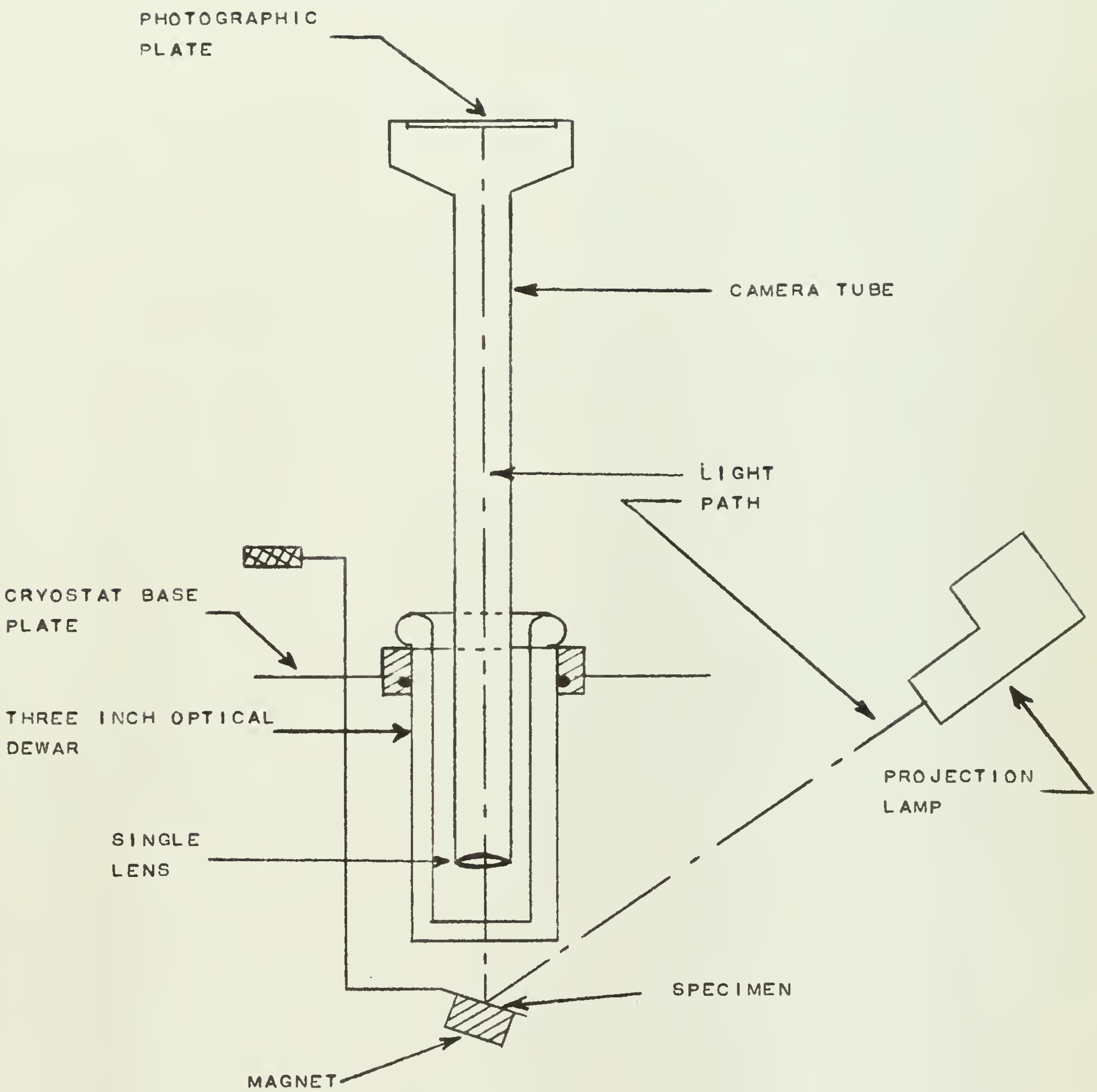


FIGURE 5 OPTICAL SYSTEM AND LIGHT PATH

TABLE I

Optical Cryostat Liquid Air - Liquid Helium Requirements.

Dewar	Coolant	Quantity	Amount Returned	Use
Six Inch Inner	Liquid Air	8 Liters	4 Liters	Precool
Ten Inch Outer	Liquid Air	15 Liters	5 Liters	Precool & Run
Six Inch Inner	Liquid Helium	10 Liters	None	Run
Six Inch Movable	Liquid Air	6 Liters	4 Liters	Cool Helium Gas

The temperature was measured by utilizing a mercury manometer and the 1949 Agreed Temperature Scale for the Vapor Pressure of Liquid Helium. Electrical connections permitted continuous monitoring of the dc resistance of the thin film during a run. The resistance of the 480 micron indium films at 27°C, is approximately ten ohms.

3. Film and Powder Techniques.

Thin indium films 480 microns wide and 1,000 Å thick were prepared by high vacuum evaporation techniques. A typical thin film specimen is shown in Figure 6. Placed in the sample holder the specimen could be moved, in a horizontal plane, from underneath the optical dewar to a position beneath the powder induction tube. Powder blown into the six inch dewar with helium gas settles down through the liquid helium onto the specimen. As indicated in section 2 above, powder was introduced at the commencement of each run before pumping on the cryostat. However, if the sample required more powder during a run, additional amounts were introduced.

Size of the niobium and nickel powder used varied from three to ten microns. For a sample width of 480 microns this gives an average ratio of powder size to sample width of 75 to one. As high a ratio as possible is desired for two reasons. The more powder particles there are per unit area of specimen, the finer and more precise will be the detail given by powder movement. A reduction in powder size is also advantageous in that particles of less mass move more easily under the influence of a magnetic field.

Originally it was planned to utilize powder size of one to two microns, but such powder was not found to be commercially available. Two types of nickel powder, one of which had a particle size of 1-13 microns and the other 7-13 microns, were obtained. The niobium had a particle size of 44 microns. It was found that this powder and the

FIGURE 6

nickel powder could be reduced in size to three to ten microns by a mechanical mortar and pestal. However, the nickel particles tended to clump together when subjected to grinding.

Introduction of powder by helium gas pressured through an atomizer has not proven to be altogether satisfactory. Control is not sufficiently precise to ensure that a uniform layer is deposited over the specimen.

4. Optical Techniques.

The optical path was designed to provide a magnification at the camera plate of 4X to 10X. The resolution capability of the lens system is on the order of five microns. The single lens in the optical system is placed at the bottom of the optical tube 10 to 20 centimeters above the sample. The photographic plate is located approximately 120 centimeters above the camera lens. The lens mounting at the lower end of the optical tube allows installation of variably sized lenses. Focus of the camera can be observed on the ground glass attachment at the top of the optical tube. Shutter speeds can be varied down to 125th of a second. Lighting for the specimen is provided by an externally located projection lamp, light intensity control being provided by a variac. The camera picture was enlarged to give an overall magnification for the system of 100X.

5. Summary.

This project terminated without realization of satisfactory results inasmuch as no domain structure was observed photographically. As anticipated the intermediate state could be readily induced either immediately below the critical temperature or the lambda point if no external magnetic field was imposed. Under the influence of a slight magnetic field partial switching could be induced only in the approximate range of 2.3° to $2.6^{\circ}K$. At all temperatures below about 2.3° and above 1.9° transition from the superconducting to normal state occurred immediately the critical current was reached. (2.6° to 2.8° is about the critical temperature for these specimens).

Failure to attain conditions appropriate for photographing domain structure is considered due primarily to inadequacies in the powder induction technique. As noted previously, control of the quantity induced was not sufficiently precise to provide the desired light uniform layer over the film.

The resolution and magnification of the optical system is considered adequate, and to provide some indication of the optical capabilities a picture of a 500 micron specimen with a light covering of niobium powder, is included as Figure 7.

Should further work on this project be contemplated, the following suggestions might prove useful:

(1) Scale down the size of the entire installation so that a four inch (or perhaps a three inch) dewar can be

FIGURE 7

used in lieu of the present six inch dewar. Such a size reduction will reduce the quantities of liquid air and helium required and render the equipment more manageable.

(2) Further the efforts to reduce powder size to the order of one micron and work with films of lesser width - perhaps 150 to 300 microns. Such films will require smaller currents to induce transition at lower temperatures. (500 micron specimens at low temperatures required currents up to 1000 milliamperes for transition.)

(3) Provide positive metering control for powder induction.

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